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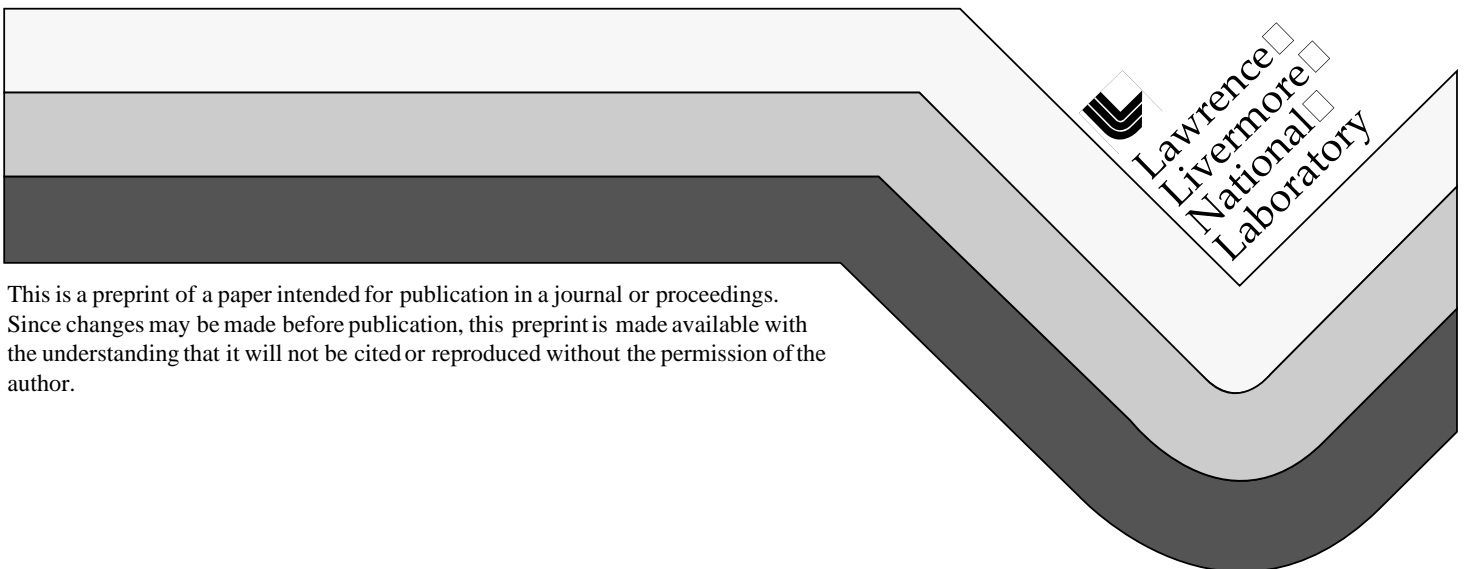
PREPRINT

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This paper was prepared for submittal to the  
SPIE's 44th Annual Meeting of the International Symposium on  
Optical Science, Engineering, and Instrumentation  
Denver, Colorado  
July 18-23, 1999

August 3, 1999



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# Smoothing of mirror substrates by thin-film deposition<sup>1</sup>

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## ABSTRACT

Superpolished optical flats with high spatial frequency roughness below 0.1 nm have been commercially available for years. However, it is much more difficult to obtain figured optics of similar quality. We have obtained and tested the finish of figured optics from different vendors by atomic force microscopy and optical profilometry and have investigated how the substrate quality can be improved by the deposition of thin films. We have determined the growth parameters of several thin-film structures. From these parameters we can determine how the surface topography of a coated mirror differs from that of the substrate, select the best thin-film structure, and predict the possible improvement.

Keywords: Smoothing films, multilayer coatings, finish of mirror substrates

## 1. INTRODUCTION

The quality of commercially available mirror substrates varies dramatically, and only one company has up to now demonstrated the capability to produce mirrors that meet simultaneously the specifications for figure and finish required for EUV lithography [1]. It has long been known that substrate quality can be improved by the deposition of an appropriate thin film. Films with graded thicknesses can be used to reduce low-frequency figure errors [2-5], while high-frequency roughness can be covered and smoothed by thin films [6]. However, thin films also generate their own intrinsic roughness, and an ideally smooth substrate always becomes rougher after film deposition. Smoothing can therefore only be expected for substrates with a roughness that is larger than the intrinsic thin-film roughness. Very smooth films with sharp boundaries have been obtained in multilayer x-ray and EUV mirrors, and these structures appear most promising for improving rough surfaces. Furthermore, the growth of these films can often be described by a simple model. Once the parameters of this model are known, one can predict theoretically how a film structure will modify the topography of a substrate, and what substrate specifications are needed for a desired imaging performance.

The growth parameters of a thin film structure were obtained by measuring the surface topography using atomic force microscopy (AFM) and optical profilometry before and after coating. These instruments cover spatial periods from about 0.5 nm to 1 mm. We found that thin films modify a substrate only for high spatial frequencies  $f > 0.01 \text{ nm}^{-1}$ , and we will present data mostly in this range.

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<sup>1</sup> Proc. SPIE 3767, 1999

Substrates were obtained from different sources with widely varying quality. The multilayer films were deposited by electron-beam evaporation with ion polishing, magnetron sputtering, or ion-beam deposition. The next section contains a short discussion of the growth model; subsequently we will describe the experimental procedure, give the results, and discuss the potential applications.

## 2. MULTILAYER GROWTH MODEL

We describe a rough surface by its 2-dimensional power spectral density  $PSD(\mathbf{f})$ , [6-10] which is a function of spatial frequency  $\mathbf{f}=(f_x, f_y)$ , where  $f=1/\Lambda_s$ , and  $\Lambda_s$  is a spatial period on the surface. During film growth, particles or atoms arrive randomly at the substrate during deposition, and this random rain of particles produces roughness with a flat white noise power spectrum:

$$PSD_2(f_x, f_y) = \Omega d, \quad \text{with } f = \sqrt{f_x^2 + f_y^2} < f_{\max}, \quad (1)$$

where  $\Omega$  is the particle volume and  $d$  the film thickness with a rms film roughness

$$\sigma^2 = 4\Omega d f_{\max}^2. \quad (2)$$

The value of the PSD is proportional to the particle volume and film thickness and the roughness increases proportional to the square root of the thickness. Most of the roughness occurs at high spatial frequencies.

Allowing the particles to relax sideways has two effects: it reduces roughness at the highest frequencies (e. g., atoms can move from a high point to a valley, and re-evaporate preferentially from high points), and a growing film can smooth out the roughness of the underlying layers. In the model of Stearns [6] the growth of a very thin layer  $i$  on top of layer  $i-1$  is expressed by

$$w_i(\mathbf{f}) = h_i(\mathbf{f}) + a_i(\mathbf{f}) w_{i-1}(\mathbf{f}), \quad (3)$$

where  $w(\mathbf{f})$  is the height distribution of the surface,  $h_i(\mathbf{f})$  is the intrinsic thickness distribution of the growing film  $i$ , and  $a_i(\mathbf{f})$  is the frequency-dependent replication factor from film  $i-1$  to film  $i$ .

$$a_i(\mathbf{f}) = \exp \left( -(\nu_i d_i q_s^n) \right), \quad (4)$$

$$l_r^{n-1} = \nu, \quad (5)$$

where  $q_s = 2\pi f = 2\pi/\Lambda_s$ . The parameter  $\nu$  is a measure for the distance  $l_r$  over which particles can relax. From (3) and (4) one obtains for the PSD:

$$PSD_2(q_s; d) = \Omega \frac{1 - \exp(-2\nu d q_s^n)}{2\nu q_s^n}. \quad (6)$$

The PSD has the value  $\Omega d$  of eq. (1) for low spatial frequencies and becomes a power law with exponent  $n$  for high frequencies. The exponent  $n$  indicates the type of growth and relaxation of the film. An exponent of 1 indicates viscous

flow; 2, condensation and re-evaporation; 3, bulk diffusion; and 4, surface diffusion [11].

The PSD of a film on a substrate is obtained as

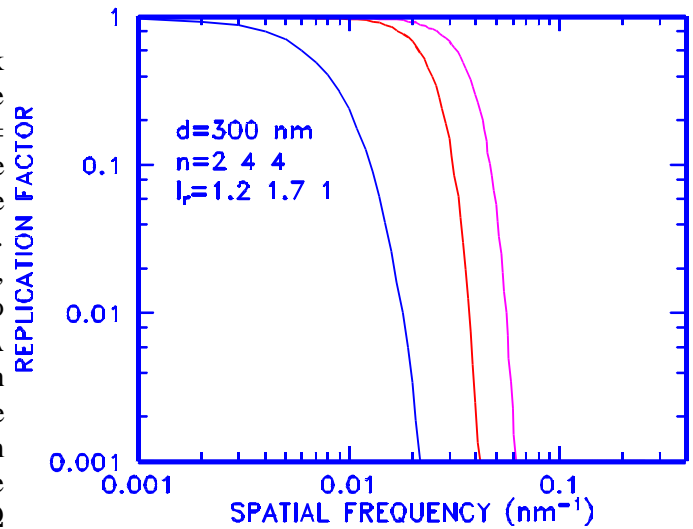
$$PSD^{tot} = PSD^{film} + a^2 PSD^{sub}, \quad (7)$$

and the roughness contributed from the range of spatial frequencies  $f_1$  to  $f_2$  is

$$\sigma^2 = 2\pi \int_{f_1}^{f_2} PSD(f) df. \quad (8)$$

The intensity of scattered light is proportional to the PSD, and the PSD can be obtained from a measurement of scattering [13].

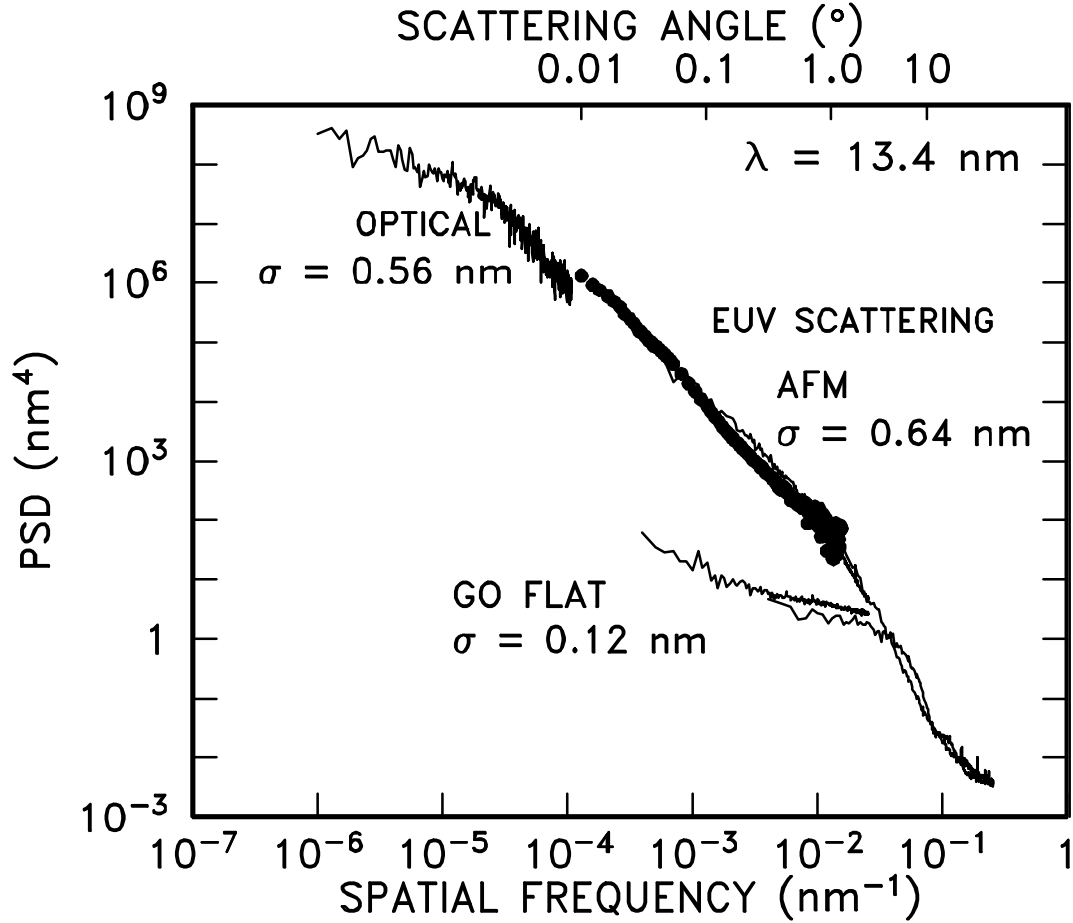
Fig. 1 shows the replication factor through a 300 nm thick film structure for some values of  $l_r$  and  $n$ . Most films have replication factors between the two right curves with  $n = 4$ . For  $f < 0.01 \text{ nm}^{-1}$  we have perfect replication of the substrate roughness to the top of the thin film, while smoothing occurs for spatial frequencies above  $0.03 \text{ nm}^{-1}$ . Thicker films can extend smoothing to lower frequencies, but one has to increase the film thickness by a factor  $2^n$  to extend smoothing by a factor of 2 to lower frequencies. A growing film reaches a steady state roughness for high frequencies that does not depend on thickness or the substrate roughness. In this region roughening by random deposition and smoothing by relaxation compensate. The coating for most efficient smoothing has low values for  $\Omega$  and large  $v$  or  $l_r$ ; a small value of  $n$  is desirable because the more gradual transition makes it easier to extend the smoothing process to lower spatial frequencies (left curve in Fig. 1) with thicker films. Some of our data indicate that it is possible to produce coatings with  $n=2$ .



**Fig. 1.** Replication factor for a 300 nm thick film with the parameters  $n=2, 4, 4$  and  $l_r=1.2, 1.7, 1$  nm for the curves from left to right.

### 3. SUBSTRATES

Figure 2 shows the measured PSD's of two good commercially obtained mirror substrates. Only one, produced by General Optics labeled GO [12] meets the EUV lithography specification. This quality is commercially available on flats that are used for laser gyros. The other mirror was used for the SANDIA 10X camera in 1997 (see Ref. 13-15) and has been characterized by AFM, optical profilometry and EUV scattering [13]. The top scale in Fig. 2 shows the scattering angle for each spatial frequency for mirrors illuminated with  $\lambda=13.4 \text{ nm}$  at normal incidence as in an EUV camera. For the mirrors of the 10X camera the total diffusely scattered intensity is about the same as the specularly reflected intensity [13]. Recently (1998) mirrors with improved finish have been obtained for the 10X camera that have reduced scattering by an order of magnitude [14, 15]. One vendor (Tinsley [12]) has now (1999) delivered a similar finish on figured aspheres for the EUV lithography project [1]. Several other vendors have delivered figured substrates within the range of the PSD's shown in Fig. 2.



**Fig. 2.** Measured PSD for two good mirror substrates measured by AFM, optical profilometry (curves) and derived from EUV scattering (points) in Ref. [13].

Most of the growth parameters have been determined from coatings deposited on supersmooth optical substrates like that labeled GO in Fig. 2. We have also deposited multilayer films on rougher substrates. Table 1 lists substrates that we have used for the electron-beam/ ion polish coating experiments. One set of 6 glass substrates of 5 cm diameter was polished in the Livermore optical shop, and each of them was characterized by AFM over square areas of  $10\ \mu\text{m} \times 10\ \mu\text{m}$  and  $1\ \mu\text{m} \times 1\ \mu\text{m}$ . The PSD and roughness were calculated from all pictures by 2-D Fourier transform [7-10]. For another set of 20 substrates of 2.5 cm diameter only 2 were characterized by AFM. The superpolished silica substrate that was coated with amorphous Si provided a mirror where roughness was more weighted to higher spatial frequencies.

The table shows that the polished substrates have considerable roughness at low spatial frequencies (column 3) where the replication factor (see Fig. 1) is close to one. We can expect smoothing by thin films only for high spatial frequencies above  $0.02\ \text{nm}^{-1}$ . Therefore smoothing will reduce only part of the roughness in column 4, and the overall reduction in roughness will be small. Furthermore we found that AFM data taken on rough surfaces are much less reliable than those from superpolished mirrors. While the uncertainty in the values of the PSD is below 50% (error in roughness below 20%) for superpolished clean surfaces, much larger variation are found for the substrates of Table 1. Repeated measurements in different areas may produce PSD values that differ by a factor of two; each picture usually contains also a few isolated features (scratches, remnants of polishing materials and other contaminations) that strongly affect the statistics of that specific image. Therefore the expected smoothing might be hidden by the noise in the data, and we have observed smoothing above noise only for the smoothest substrates of Table 1 (see Fig. 3 below). AFM data are also influenced by contamination of the surfaces and careful cleaning of the surfaces is important. Data from the top surface of a coating should be taken immediately after coating to minimize the effect of contamination and

reduce the influence of any chemical reaction on the surface.

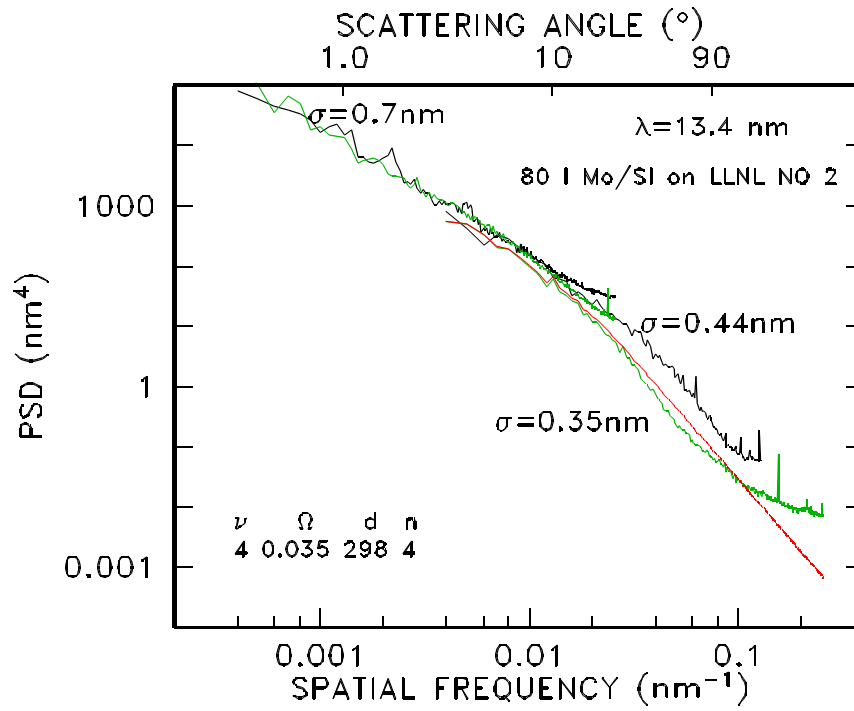
In addition to these relatively rough substrates, we used superpolished optical flats from General Optics [12], 1 inch and 2 inch squares of float glass, Si wafers and Zerodur substrates. Some of these substrates have roughness in the 0.1 nm region, and the performance of the coated mirrors is for them mostly determined by the thin film growth. The growth parameters of thin films can much more reliably be determined from coatings on good substrates, and we mostly relied on these data for our analysis.

**Table 1.** Roughness values of different substrates derived from 10 $\mu$ m x 10 $\mu$ m and 1 $\mu$ m x 1 $\mu$ m AFM pictures.. The last substrate is amorphous Si on superpolished fused silica. The last row indicates the frequency ranges in the PSD over which the roughness was obtained using eq. (8).

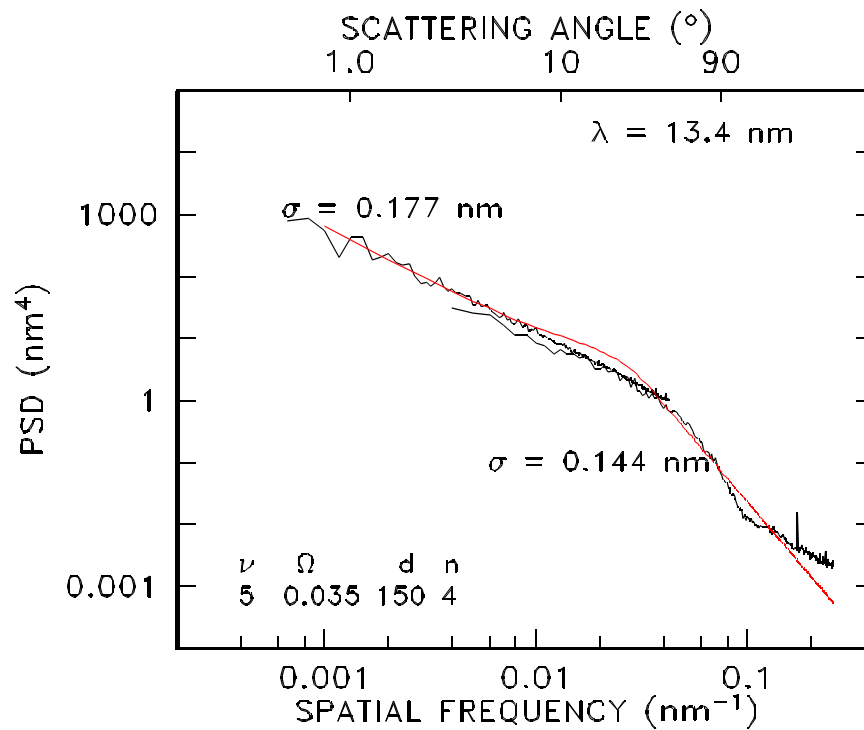
Substrate	Dia (cm)	$\sigma_l$ (nm)	$\sigma_h$ (nm)
LLNL NO1	5	1.07	0.68
LLNL NO2	5	0.70	0.44
LLNL NO3	5	1.95	1.15
LLNL NO4	5	0.53	0.35
LLNL NO5	5	1.46	0.94
LLNL NO6	5	1.76	0.79
LLNL 1	2.5	0.45	0.21
LLNL 2	2.5	0.81	0.39
15 nm aSi	2.5	0.21	0.31
frequency range		0.0004-0.0256 nm <sup>-1</sup>	0.004-0.128 nm <sup>-1</sup>

#### 4. MULTILAYER DEPOSITION AND CHARACTERIZATION

The substrates of Table 1 were coated with multilayers by electron beam evaporation with *in-situ* reflectivity monitoring and ion-beam polishing [17-22]. Most of the mirrors on these substrates are Mo/Si with periods around 7 nm. The Si layers were deposited about 3 to 4 nm thicker than required and then back etched with the ion beam to the desired thickness. The ion beam intensity was not uniform over the rotating substrate holder, therefore samples with different degrees of ion-polishing could be produced in one deposition run. The Mo/Si mirrors with less polishing have therefore larger periods. In addition to the substrates of Table 1 we coated also several smooth, superpolished quartz and Si wafers in each deposition run. Growth parameters were obtained by fitting the PSD's obtained by AFM to the growth model; in addition reflectivity data near normal incidence with EUV and at grazing angles of incidence at  $\lambda=0.154$  nm were obtained for all samples and gave an independent determination of the quality of the boundary.

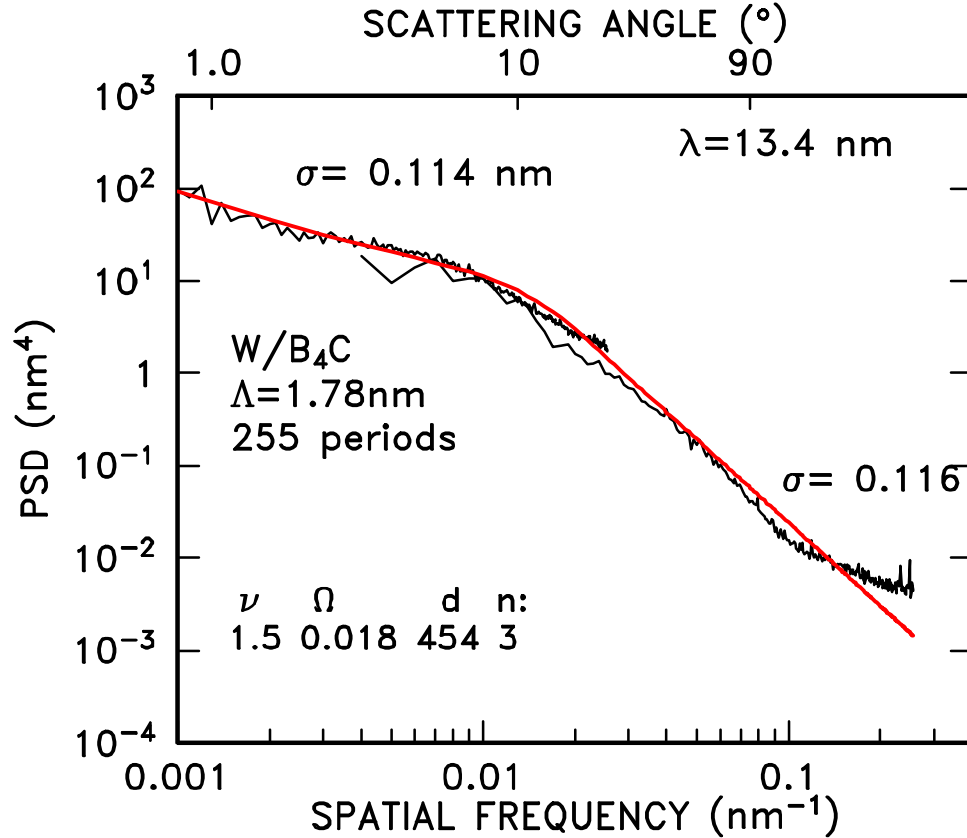


**Fig. 3.** Measured PSD's of the uncoated substrate LLNL NO 2, of the top of a 80 layer Mo/Si structure on it, and calculated PSD of the top of the multilayer.



**Fig. 4.** Measured and calculated PSD's for a 30 period multilayer on float glass with the growth parameters of the coating.





**Fig. 5.** Measured and fitted PSD of a short period W/B<sub>4</sub>C multilayer on Si (sample courtesy of Y. Platonov)

Fig. 3 shows the measured PSD of one of the mirrors in Table 1 before and after coating it with a 80 layer Mo/Si structure. The PSD obtained from the 10  $\mu\text{m}$  pictures is not changed by the coating, with the roughness remaining at 0.7 nm, while we notice a reduction in roughness in the 1  $\mu\text{m}$  pictures from  $\sigma=0.44$  to  $\sigma=0.35$  nm. The smooth curve in the figure shows the calculated PSD using eqs. (6) and (7) and the growth parameters shown in the figure. The growth parameters used are in agreement with those obtained from smooth substrates and values in the literature [8].

An example for a PSD of the top of a Ni/C multilayer on a smooth floatglass substrate is shown in Fig. 4. As before we show the data obtained from a 10  $\mu\text{m}$  and a 1  $\mu\text{m}$  image. The smooth curve shows the fit with the growth parameters given in the figure. For the fit we assumed that the low frequency PSD is that of the substrate and extrapolated the low frequency PSD to high frequencies using the power law  $\text{PSD}=0.0025 f^{-1.8}$  for the substrate. Eqs.(6) and (7) produce the smooth curve.

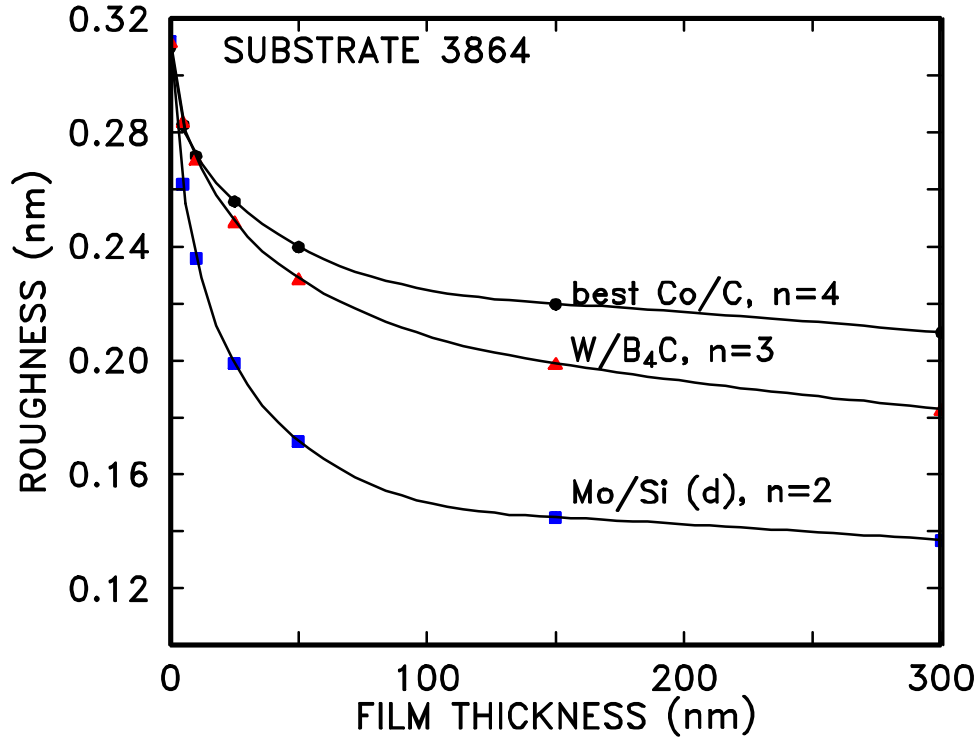
The measured and fitted (smooth curve) PSD's of a W/B<sub>4</sub>C of 255 periods and period thickness of 1.78 nm is shown in Fig. 5. The substrate is a Si wafer and the PSD of the substrate is assumed as  $\text{PSD}=0.03/f^{1.15}$ . It is remarkable that the power spectrum of the coating has a high frequency power law with exponent 3. In all three figures smoothing reduces the high frequency roughness and can be recognized in the change of the slopes of the PSD's. For frequencies above the knee in the PSD's we have effective smoothing and the smoothing factor can directly be recognized in Fig. 3-5 as the difference in the extrapolated low frequency PSD and the actual PSD. The substrates of Table 1 have considerable roughness at low frequencies that are replicated, and therefore the overall roughness is only slightly reduced by the film smoothing.

Substrates that have roughness mostly at high frequencies can be smoothed more effectively by thin film deposition. We have obtained such a substrate by coating a Si wafer with a 15 nm thick film of amorphous Si by magnetron sputtering in argon at 10 mTorr pressure. This film has considerable high frequency roughness, and we can remove some of this roughness by an additional multilayer coating. Fig. 6 shows how the roughness of this substrate decreases when it is overcoated with three of the multilayer systems of Table 2. With the best Mo/Si system we can reduce roughness to 0.13 nm and bring the mirror into a range that would be acceptable for EUV cameras.

A summary of the growth parameters of all coatings tested is given in Table 2. Most coatings are described by a value of  $n = 4$  and have replication factors between the two right curves in Fig. 1. The Mo/Si coating (d) with  $n = 2$  can extend smoothing to lower frequencies as represented by the left curve in Fig. 1 and demonstrated in Fig. 6. Low values of  $n$  have also been reported for W/Si multilayers [22]. The last column in Table 2 shows the roughness of the thin film structures with the given parameters on a perfectly smooth substrate. These roughness values would also be obtained on substrates that have all their roughness at very high spatial frequencies  $f > 0.04 \text{ nm}^{-1}$ .

**Table 2.** Growth parameters of multilayer systems with multilayer period  $\Lambda$ , number of periods  $N$ , growth parameters  $\Omega$ ,  $l_r$ , and  $n$ , total thickness  $d$ , and roughness  $\sigma$  of the multilayer film calculated for a perfectly smooth substrate within the spatial frequency range  $f = 0.001 - 0.25 \text{ nm}^{-1}$ . Coating (a) produced by dual ion beam sputtering courtesy of J. Pedulla (NIST), (b) of Y. Platonov (Osmic). Data for Mo/Si produced by magnetron sputtering (c) from Ref. [8], and ion beam sputtering (d) from P. Kearney and D. Stearns (LLNL). Parameters are the average value for both coating materials, for sputtered Mo/Si we give also the parameters for Mo and Si separately.

System	$\Lambda (\text{nm})$	$N$	substr.	$\Omega (\text{nm}^3)$	$l_r (\text{nm})$	$n$	$d (\text{nm})$	$\sigma (\text{nm})$
Co/C	3.2	150	floatglass	0.016	1.44	4	480	0.14
Co/C	2.9	144	GO	0.016	1.71	4	423	0.12
Co/C	2.4	144	GO	0.016	1.14	4	342	0.15
Co/C	3.2	85	floatgl	0.016	1.71	4	274	0.19
Mo/Si	7.2	24	LLNL1	0.035	1.71	4	172	0.14
Ni/C (a)	5.0	30	floatglass	0.035	1.71	4	150	0.14
W/B <sub>4</sub> C (b)	1.22	350	Si	0.01	1.22	4	427	0.10
W/B <sub>4</sub> C (b)	1.78	255	Si	0.018	1.22	3	454	0.12
Mo/Si (c)	6.8	40	Si	0.035	1.36	4	280	0.19
Mo/Si (d)	6.8	40	Si	0.055	1.20	2	280	0.12
Si in Mo/Si (c)	6.8	40	Si	0.02	1.36	4	280	0.14
Mo in Mo/Si(c)	6.8	40	Si	0.5	1.36	4	280	0.23



**Fig. 6.** Calculated roughness of coatings on the rough amorphous Si substrate of Table 1 as a function of total film thickness for smoothing films of Co/C, W/B<sub>4</sub>C, and Mo/Si with  $n=2$ . Growth parameters are taken from Table 2. Substrate PSD =  $2 \cdot 10^{-6} / (0.035^2 + f^2)^{2.5}$  in nm<sup>4</sup>. Roughness for the  $f = 0.001 - 0.256 \text{ nm}^{-1}$  range.

## 5. DISCUSSION

A perfect, smooth substrate can only be roughened by the addition of a thin film. Only rough substrates can be improved, and smoothing occurs only for very high spatial frequencies or very short periods, below about 50 nm. The substrates that we received from optical companies that did not meet the requirements for EUV imaging, had in most cases considerable low frequency roughness, and thin film smoothing does not bring them within the specifications of 0.1-0.2 nm roughness. The relaxation lengths of all our thin film structures are in the 1-2 nm range, corresponding to a sideways relaxation over about 5 atom sizes. It seems improbable that this range can be dramatically improved by changing deposition parameters. The strengths of the bond of an atom can be affected by local curvature, and the deposition rate might be affected locally by the surface slope. Both quantities are extremely small when we consider roughness in the 1 nm heights range with a spatial period in the 1  $\mu\text{m}$  range. Deposition processes where the relaxation is dominated by condensation/reevaporation with  $n = 2$  have a more gradual transition from replication to smoothing and extend smoothing to lower spatial frequencies than those with larger  $n$ . One system (W/Si) with low  $n$ -value has been described before [16]. Relaxation by viscous flow with  $n=1$  would produce the most efficient smoothing possible with our growth model. We have seen no indication of such a process in any of our samples; however, the smoothness of float glass and lacquer coated surfaces could be explained by viscous flow. It might be worthwhile to look for deposition processes where a liquid phase occurs on the film surface for some fraction of the deposition time. In some cases it might be possible to fit the measured data with different sets of growth parameters than we have given in Table 2 or use a superposition of different relaxation processes to fit the data. For example the slope of the measured PSD's in Figs 3 and 4 is higher than the fitted curves in the region around  $0.05 \text{ nm}^{-1}$ . We assumed that this is caused by a loss in contrast in the AFM, and we further assumed that the higher values around  $0.2 \text{ nm}^{-1}$  are instrument noise. Independent measurements on these samples similar to the scattering data of Ref. [16] could be used to check these assumptions. However, independent of the details of the fit, we can observe the transition from replication to smoothing

in the  $0.01 - 0.02 \text{ nm}^{-1}$  range as the change in the slope of the measured PSD and can state that smoothing occurs for  $f > 0.02 \text{ nm}^{-1}$ .

All film structures investigated so far were multilayers optimized for the highest EUV or x-ray reflectivity. It is well known that the roughness of these films usually oscillates during the deposition, with one of the components, usually the spacer layer, having a smoothing, the other a roughening action [17-22]. Making the smoothing film (the C or Si layer) thicker and the roughening film thinner could result in more efficient smoothing of the substrate and has still to be investigated. A limitation for this process will be the increased film stress and the possibility of stress induced roughening.

Multilayers that use only the smoothing "spacer layers" might be more suitable for substrate smoothing than the systems we have investigated. In order to obtain good reflectivity it is important that all boundaries within a multilayer are sharp without diffusion of one material into the other. This requirement is not needed for smoothing films; only the quality of the top surface is important. Diffusion of the two film materials into each other might lead to an additional relaxation process and produce films with better smoothing capabilities than we have observed up to now.

## 6. SUMMARY

We have determined the growth parameters of several multilayer structures and explored if these structures can be used to smoothen mirror substrates. We find that we can reduce high frequency roughness with spatial periods  $f > 0.02 \text{ nm}^{-1}$  to values in the  $\sigma = 0.1 - 0.2 \text{ nm}$  range. The roughness of mirror substrates for these frequencies would not affect the performance of a coated mirror. We have measured the roughness spectrum of mirror substrates obtained from different sources for application in EUV lithography or microscopy and found that those with large roughness ( $\sigma > 0.4 \text{ nm}$ ) had always considerable low frequency roughness that is replicated in the multilayer films. Our thin film structures can not reduce roughness of these substrates sufficiently to bring them within the specification of EUV optics. Substrates that have been roughened by thin film deposition often have roughness at very high spatial frequencies, and this roughness can be removed with an overcoating of the appropriate multilayer structure.

## ACKNOWLEDGMENTS

We thank D. Stearns and E. Gullikson for many discussions and access to their data. The substrates of Table 1 were provided by J. Taylor, the rough Si coated substrate by D. Stearns and F. Weber, and P. Kearney fabricated the coating (d) of Table 2. The W/B<sub>4</sub>C samples were obtained from Y. Platonov and the Ni/C multilayers from J. Pedulla. The work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract No. W-7405-ENG-48. Funding was provided by the Extreme Ultraviolet Limited Liability Company (EUV LLC) under a Cooperative Research and Development Agreement and by NSF grant MPS-9612204.

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